Packaging and Reliability of Electronic Nose for Space Applications

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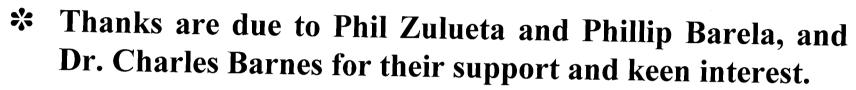
> Rebecca Young Kennedy Space Center, Orlando, FL

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OUTLINE

- *Objectives
- *Introduction
- *Types of Sensors
- **★JPL E-Nose test results**
- *Reliability issues
- *****COTS E-Nose program
- *Packaging and Qualification
- *Conclusion Assess and Validate JPL E-Nose and COTS e-noses via collaboration within NASA and outside

Packaging and Reliability Assessment of Electronic Noses

- An electronic nose is to identify an odorant sample and perhaps to estimate its concentration.
- COTS E-Noses and JPL E-Nose are being used for such applications.
- The e-nose could monitor indoor air quality as well as industrial and agricultural, and space shuttle cabins, and cabins of international space station such as hazardous alarms for toxic (ammonia, monomethylhydrazine, hydrazine, aromatic gas species, and inorganic gas species), biological agents etc.
- An electronic nose could be used to monitor electrical fires in control equipment and also high power electronics packaging systems for early detection of fire.
- We will address the assessment of reliability of e-nose sensor heads and related driver electronics packages of the respective e-nose to meet manufacturer's specifications and also assess beyond manufacturer's specifications that include to meet the requirements of NASA.



Electronic Nose

POSSIBLE APPLICATIONS





SPACE STATION ENVIRONMENTAL MONITORING Event monitor for early warning of spills, leaks, fires; clean-up monitoring; automated environmental control.



MILITARY APPLICATIONS
Air quality monitor for enclosed spaces.
Detection of explosives, chemical/biological warfare agents.



INDUSTRIAL MONITORING AND PROCESS CONTROL Monitor food processing, identity and condition of raw materials, leaks and buildup of toxic compounds.



MEDICAL
Diagnosis of diseases with characteristic odors, monitor patients' rooms, monitor labs.



OTHER ENVIRONMENTAL MONITORING
Air quality in buildings, aircraft. Presence of toxic materials in designated spaces.



Rationale to NASA



 No miniature gas sensors capable of detecting and identifying a broad suite of compounds



- ◆ GC-MS are capable of highly accurate analysis at very low levels require significant crew time to operate and maintain
- ◆ Lack of a monitor in crew quarters of space shuttle or space station
- ◆ Need of such capability is foreseen
- ◆ Air in the space station cannot be easily replaced
- ◆ Contaminants will build up over time
- ◆ Air quality should be monitored since crew health is important
- ◆ New technology infusion to create an integrated environmental monitoring and control system for the ISS and other missions.

 Ramesham

JPL Definition

- ◆ Electronic noses are systems for the automated detection and classification of odors of simple and complex and it is generally composed of an array of chemical sensing systems and a pattern recognition system. Electronic noses are based on human olfactory system. (Gardner, 1988)
- ◆ Arrays of sensors, which respond to a wide range of compounds. (Chemical sensors, spectrometers)
- ◆ Advanced pattern recognition and artificial intelligence techniques, which enables to extract relevant and reliable information.(statistical methods such as principal component analysis, discriminant factorial analysis, cluster analysis or artificial neural networks)

Ramesham



SENSES

- ◆Smell (Nose, Odor, good, bad, etc)
- ◆Taste (Tongue, sweet, spice, sour, etc.)
- ◆Feel (Touch, hard, soft, warm, cold)
- ◆Listen (Ears, sound)
- ◆See (Vision, Eyes, colors)







Objectives

- ◆ Develop, build, and demonstrate a miniature gas sensor, an E-Nose, for experiment on a shuttle flight (Accomplished by JPL)
- Assess the air quality monitor in crew habitat on a spacecraft
- ◆ Assess and validate various e-noses
- ◆ Establish damage process and failure of e-nose
- Early failure mechanism by environmental storage and thermal cycling tests
- Packaging Reliability and E-Nose reliability



Introduction



- ◆ Electronic Nose, New Technology Infusion on Space Shuttles, International Space Station (ISS)
- ◆ Demonstrated the operation of polymer based sensors based on Dr. Nathan Lewis of Caltech for use in sensing arrays
- ◆ E-Nose was flown on STS-95 to determine its utility as an air quality/incident monitor in crew habitat on a spacecraft
- ◆ JPL has developed, built and demonstrated a low power, miniature gas sensor which has capability to identify various gas species present in the recirculated breathing air of the space shuttle.

JPL

Introduction



- ◆ Constituents of breathing air in a closed chamber in which air is recycled (Space station, space shuttle, etc.)
- ◆ Air-quality is determined by collecting the samples and analyzing them on the ground using GC-MS
- ◆ Miniaturized e-nose capable of identifying contaminants in the breathing environment at ppm or ppb levels would greatly enhance the capability for monitoring the quality of recycled air as well as providing notification of presence of dangerous gases.
- ◆ An e-nose is an array of non-specific chemical sensors, controlled and analyzed electronically, which mimics the action of nose by recognizing patterns of response to vapors
- ◆ JPL/NASA needs such e-noses

JPL TYPES OF SENSORS

- ◆Conductivity sensors (metal oxide and polymer)
- ◆Piezoelectric sensors
- **◆**MOSFET
- **♦**Optical
- **♦** Spectrometry
- **◆**Electrochemical
- **◆**Chemical



Electronic Nose



ELECTRONIC NOSE FOR SPACE STATION

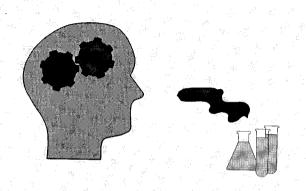
- + Incident monitor real time air quality monitoring
- Identify and quantify target compounds at SMAC level
- + Low mass, low power device
- + Requires little crew time for maintenance and calibration

	Analysis Time (min)	Concentration of Constituents	Discrimination of Constituents
ENose	.5 - 15	0.01 - 10,000 ppm	good for target set
GC-MS	10 - 100	< 10 ppb	very good
VOC	1- 5	.1 - 2000 ppm	poor
FID	1 - 5	.1 - 50,000 ppm	poor
Smoke Alarm	.5 - 5	1 - 10 ppb	none



Electronic Nose REAL TIME AIR QUALITY MONITORING



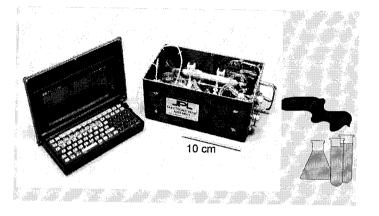


HUMAN NOSE

- array of thousands of sensors
- broad band capability
- trainable to new odors
- data acquisition in the brain
- analysis by true Neural Network processing; pattern recognition

LIMITS ON HUMAN NOSE

- fatigue
- becoming accustomed to an odor
- ♦ insensitivity to some compounds
- toxicity of some contaminants.



JPL/CALTECH ELECTRONIC NOSE

- array of a few tens of sensors
- thin film polymer based sensors
- broad band capability
- polymers selected to respond to particular compounds
- ◆ trainable to new analytes
- data acquisition by computer
- data analysis by computational methods and pattern recognition

LIMITS ON ENOSE

 insensitivity to compounds; can be overcome by sensor film selection

Electronic Nose



WHAT IS AN ELECTRONIC NOSE?

An array of non-specific chemical sensors, controlled and analyzed electronically, which mimics the action of the mammalian nose by recognizing patterns of response

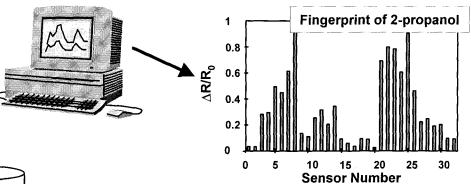
1. ENose measures background resistance in each sensor and establishes R₀.

2. Contaminant comes in contact with sensors on the sensing head.

insulating polymer conductive medium

3. The sensing media, polymer films loaded with a conductive medium such as carbon black, change resistance by swelling or shrinking as air composition changes.

4. Resistance is recorded by a computer, the change in resistance is computed, and the distributed response pattern of the sensor array is used to identify gases and mixtures of gases



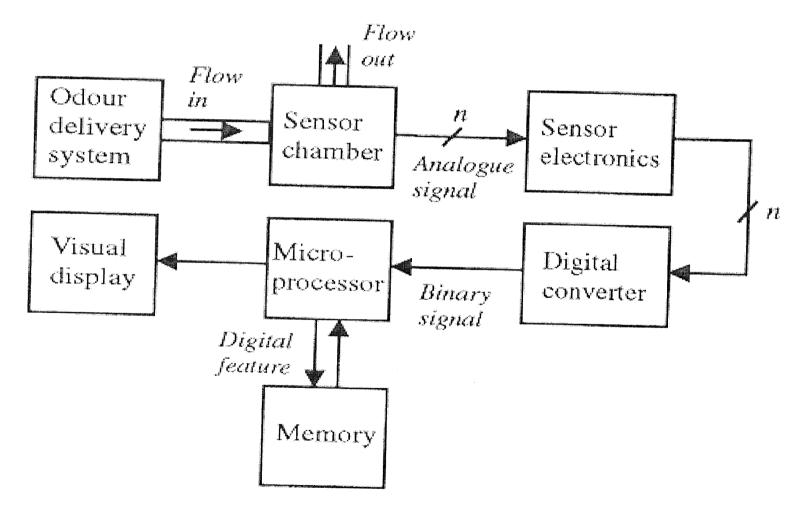
5. Responses of the sensor array are analyzed and quantified using software developed for the task.

2-propanol, 300 ppm





Basic components of an electronic nose instrument system



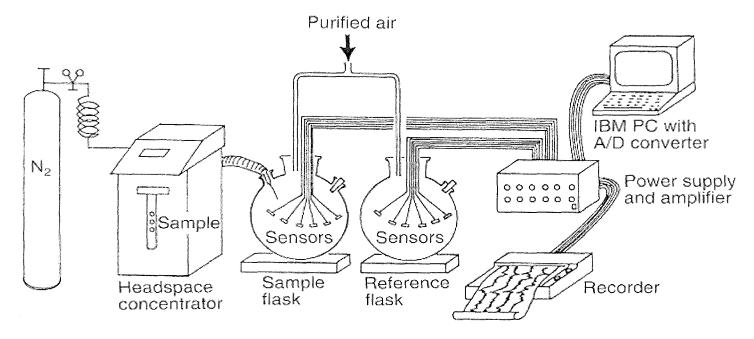
Ref.: Gardner and Bartlett





Electronic Nose Instrumentation

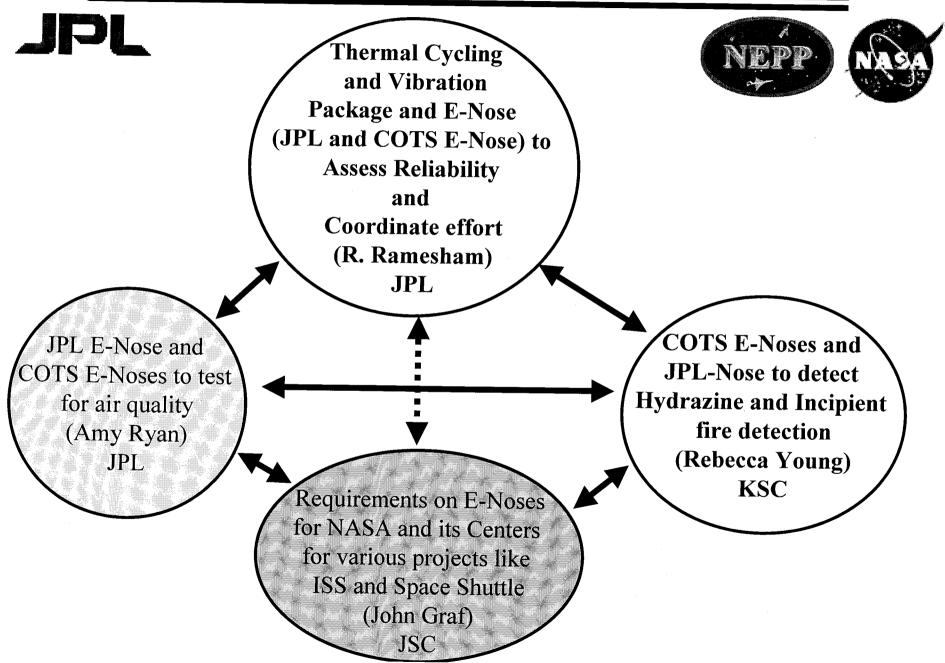
 Head space sampling and Flow injection (carrier gas may be used to carry the odorant)



Inexpensive, forced mixing; Laborious and time consuming, temperature of odour and odour sensor

Ref.: Aishima 1991

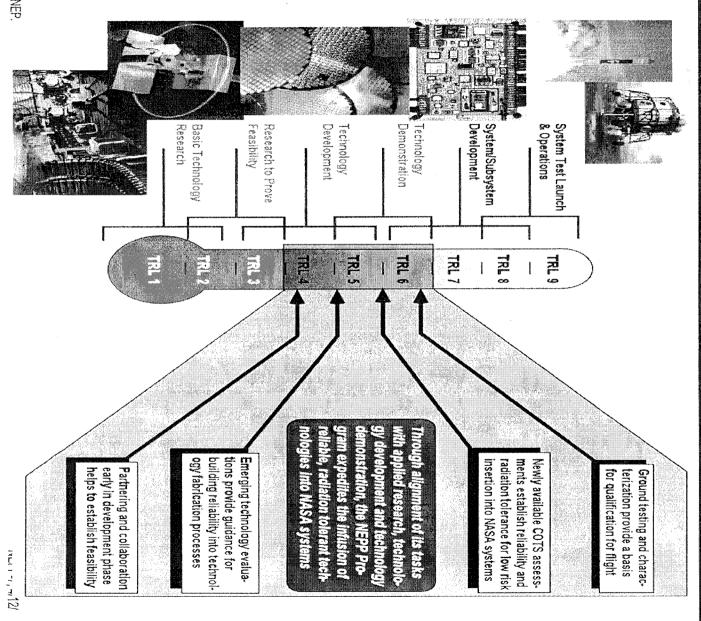
Packaging and Reliability Assessment of Electronic Noses





NEPP and TRLs: A Path to Rapid Technology Infusion





JPLCOTS E-nose companies and Odour Sensors



- Tekmar Co, HP, Sensidyne, Cyrano Sciences, Mosaic Industries, St. Croix Sensory Inc., Agilent Technology, Scientist Illumina, Inc., USA
- Figaro Engineering Inc., National, and New Cosmos Electric, Japan
- Aromascanner, Cogent Ltd., UK
- Bloodhound, UK
- Mastiff Electronic Systems, UK
- Neotronics, UK (e-Nose 4000 and 5000)
- Alpha MOS and Europhor Instruments, France
- HKR Sensor System, MoTech, Karlusruhe Research Center, Germany
- Lennartz Electronics, Germany
- Nordic Sensor Technology, Sweden
- Array Tec
- Electronic Sensor Technology, LP
- 30 50 universities around the world (Caltech, JPL, NC state, etc.)





World-wide market size and value of electronic noses

Year	Estimated number of units sold	Market value (\$ Million)
1993	5	0.32
1994	25	1.6
1995	100	6.4
1996	250	16.0
1997	500	32.0
2000	2500	160.0

JPL COTS E-Nose Program in Summary

- ❖ Single set of reliability testing requirements for a wide applications may not be possible for evaluation of E-Nose technology
- ❖ Finding a common denominator and standardized testing based on the E-Nose key failure mechanisms are valuable to user community
- User can carryout any additional reliability testing needed for their application
- Standardized test methodology will reduce unclear communication between users and suppliers

JPL COTS E-Nose Program in Summary

- ❖ Start with high volume COTS type E-Nose components that have potential for high reliability application
- ❖ Their availability and lower cost, a large number of these components can be tested to generate statistically meaningful reliability data.
- ❖ JPL has initiated COTS E-Nose Program with objectives of understanding quality and reliability assurance associated with implementation of this technology and help to build needed infrastructure.



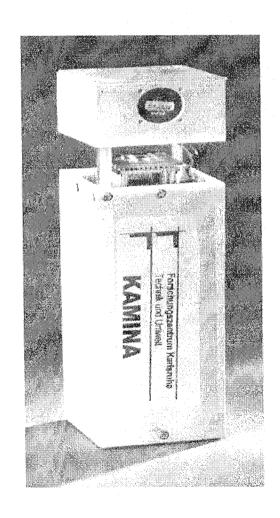
Evaluation of E-Noses and Respective IC Package



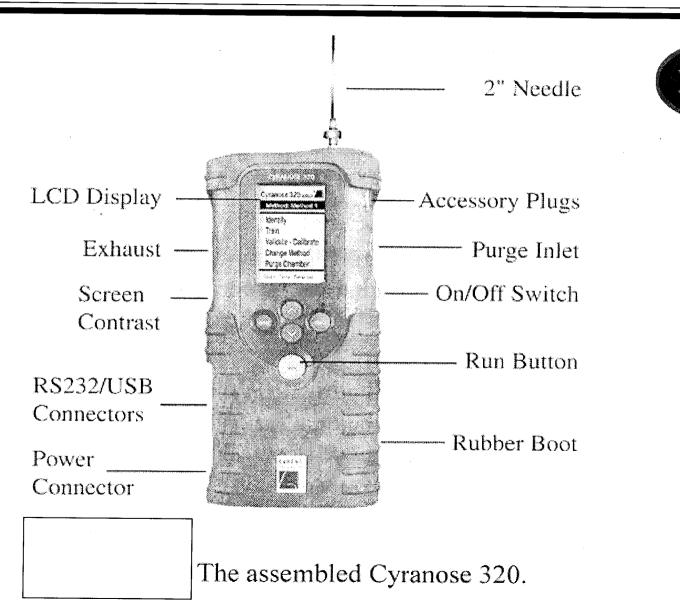


- * JPL E-Nose
- * KAMINA
- **SAM Detect**
- **Cyranos's E-Nose**
- * WMA Airsense PEN2 E-Nose

Demonstrator for sensitive detection and discrimination of overheated wire insulations based on the KAMINA technology



Packaging and Reliability Assessment of Electronic Noses





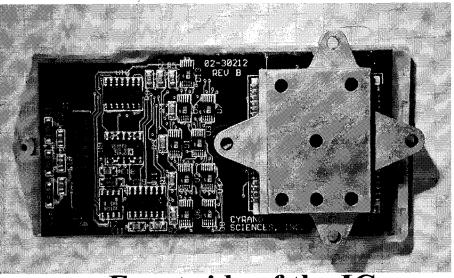
Cyranose 320



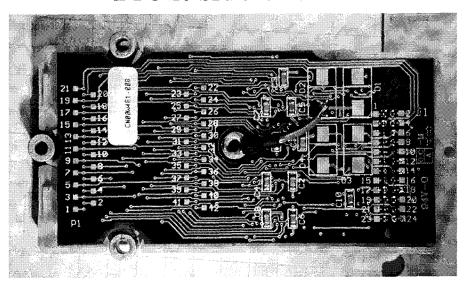




Cyranose 320



Front side of the IC



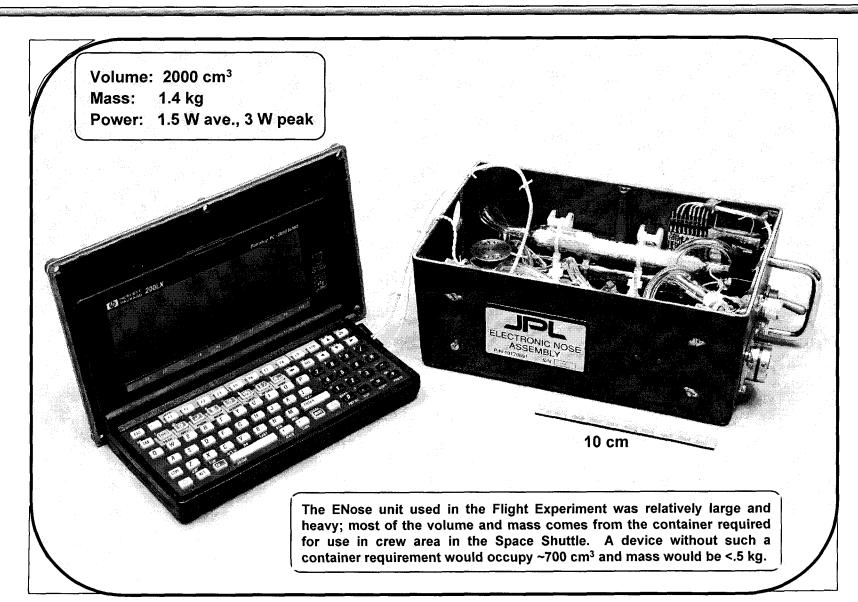
Schematic of E-Nose



Electronic Nose

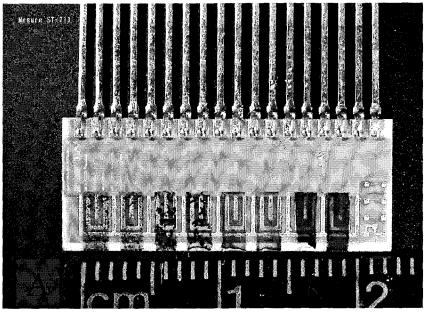


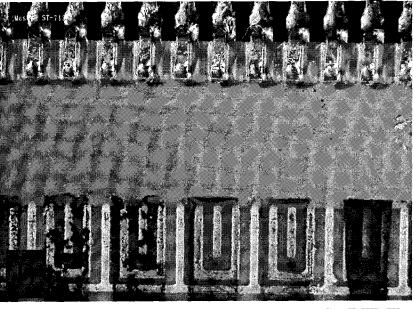




JPL

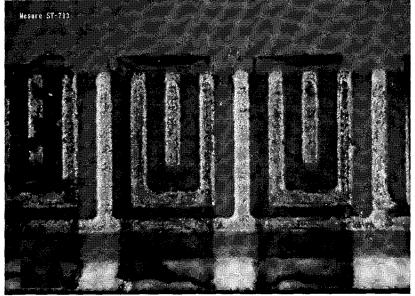






JPL E-Nose Sensor Array
A: Complete Sensor array
B: Magnified view of A
showing sensors
and interconnects
C: Magnified view of B

showing the sensors

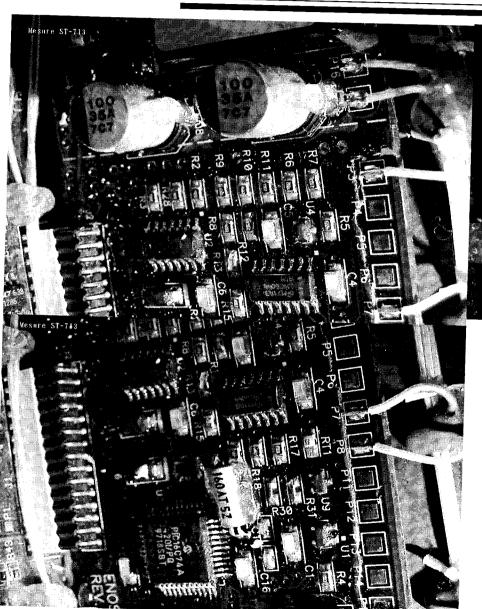


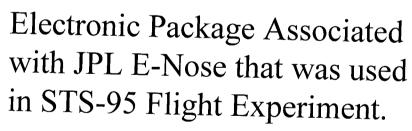
Ram

JPL E-Nose IC Package







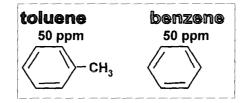


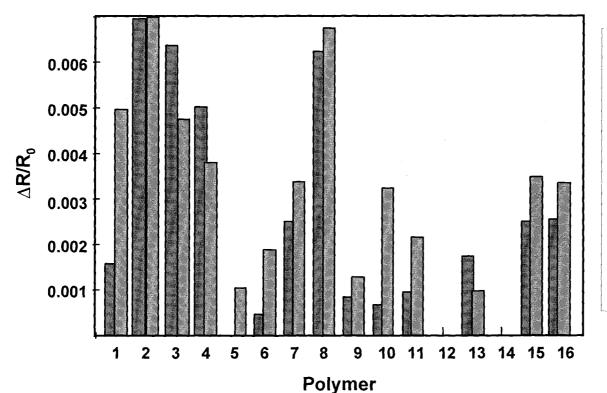


Electronic Nose

FINGERPRINT RESPONSE OF ARRAY







- 1 Poly(2, 4, 6-tribromostyrene)
- 2 Poly(4-vinylphenol)
- 3 Poly(ethylene oxide)
- 4 Polyamide resin
- 5 Cellulose triacetate
- 6 Poly(2-hydroxyethyl methacrylate)
- 7 Vinyl alcohol/vinyl butyral, 20/80
- 8 Poly(caprolactone)
- 9 Poly(vinylchloride-co-vinyl acetate)
- 10 Poly(vinyl chloride/acetate) 90/10
- 11 Poly(vinyl acetate)
- 12 Poly(N -vinylpyrrolidone)
- 13 Styrene/isoprene, 14/86 ABA
- 14 Poly(vinyl stearate)
- 15 Methyl vinyl ether/ maleic acid 50/50
- 16 Hydroxypropyl methyl cellulose, 10/30

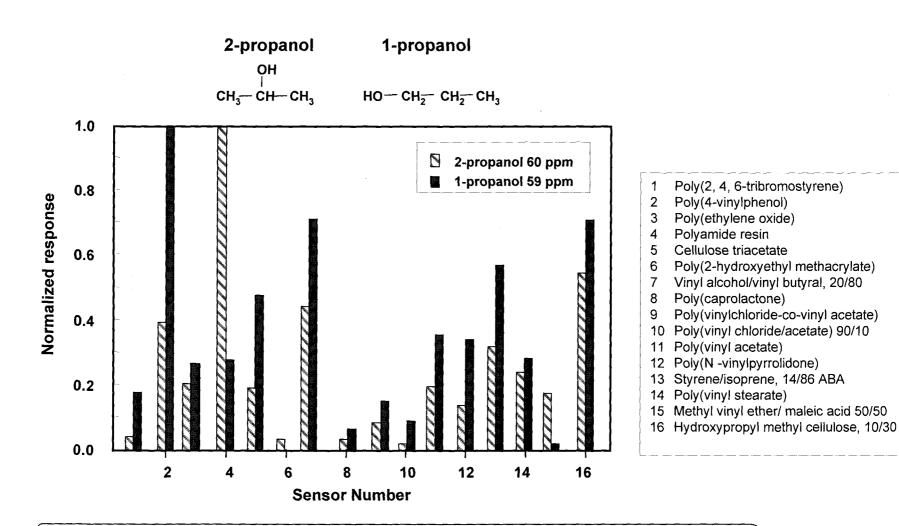
Similar compounds can be distinguished by their fingerprints. Benzene and toluene are both aromatic, and have similar but distinguishable response patterns.



Electronic Nose



RESPONSE PATTERNS OF ISOMERS



Isomers of propanol have similar but distinguishable response patterns.





Detection of simple odors by E-nose

Simple odor	No of odors	No/Sensor type
Alcohol's, ketones, alkanes, alkenes, sulfide	s 11	12/SAW
Pungent, ethereal, minty	47	7/MOS
Methanol, ethanol, propanol	3	3/MOS
Alcohol's	5	12/MOS
Alcohol's, terpenes, pyrazines	24	6/MOS
Methanol, ethanol, propanol	3	5/CP
Water, propanol, ethyacetate, acetone	4	3/CP
Alcohol's	5	20/CP
Toluene, n-octane (and mixtures)	2	6/BAW



Assess COTS E-Noses and JPL E-Nose for International Space Station (ISS) at 1 atmosphere pressure, 30-50 % relative humidity, and at a temperature of 25-35 °C.

Assess the reliability of E-Nose for several days (~15 days)

Detect the compounds at or below 1 hour SMAC or choose 3 or 4 compounds in the target list as shown in Table 1 at or below 1 hour SMAC.

Target compounds are Toluene, Ammonia, 2-propanol, Methanol, Hydrazine, and outgassing from overheating of electrical wires used on board of Space Shuttle and International Space Station.

Assess/Evaluate the COTS E-Noses for 1 ppm hydrazine in an airlock at 5 psi in about 3 minutes.

Assessment of thermal degradation events in space shuttle. Wire insulation on flight approved materials list where systems are most likely to overheat.





Target compounds for electronic nose shuttle experiment and JPL limits of detection.

Compound	Detected on the Shuttle, ppm	1 hr SMAC, ppm	Detection Limit ppm	Testing, ppm
Alcohols				
Methanol	<1	30	5	5-300
Ethanol	0.5 - 5	2000	50	10-130
2-proipanol	0.4-4	400	50	30-160
Ammonia	0	30	20	10-50
Benzene	<0.1	10	10	10-150
CO_2	320	13000		
Indole	0	1	0.03	0.006-0.06
Hydrazine	0	4	???	???
Methane	1-10	5300	3000	1000-7000
Formaldehyde	0	0.4	25	25-510
Freon 113	0.1 - 1	50	20	20-600
Toluene	0.4 - 4	16	15	15-60

Electronic nose sensor technologies

Sensor type	Principle of	Fabrication	Availability	Sensitivity
	operation	methods		
Metal oxide	Conductivity	Microfabricated	Several commercial	5-500 ppm
			types	
Conducting	Conductivity	Microfabrication	Several commercial	0.1 - 100 ppm
polymer		Electroplating	types	
		Screen printing		
Quartz crystal	Piezoelectricity	Screen printing	Several commercial	1 ng mass
microbalance		Wire bonding	types	change
		Microelectromecha		
		nical systems		
		(MEMS)		
Surface acoustic	Piezoelectricity	Microfabrication	Several commercial	1 pg mass
wave (SAW)		Screen printing	types	change
MOSFET	Capacitive charge	Microfabricatiojn	Several commercial	ppm
	coupling		types	
Gas	Molecular spectrum	MEMS, precision	Several commercial	Low ppb
Chromatography		machining	types	
Mass	Atomic mass	MEMS, precision	Several commercial	Low ppb
spectrometry	spectrum	machining	types	

Commercial Electronic Noses Available to-date in the Market

Company Name	Website	Product name	
Agilent Technologies/Hewlett-Packard	http://www.agilent.com/Top/English/index.html	HP4440A Chemical Sensor	
		HP4440B, Chemical Sensor	
Cyrano Sciences, Inc.	http://cyranosciences.com/	Cyrano 320	
Electronic Sensor Technology "zNose"	http://www.estcal.com/	4100 Fast GC Analyzer	
		7100 Fast GC Analyzer	
Microsensor Systems Inc.,	www.sawtek or www.microsensorsystems.com	VaporLab	
Alpha-Mos	http://www.alpha-mos.com/	FOX 4000, FOX 2000, FOX 3000, FOX 5000, Prometheus, Kronos, Centauri, ASTREE, Odor Scanner 100	
Lennartz-Electronic	http://www.lennartz-electronic.de/Pages/English/Homepage_english.html	Moses II	
Motech Sensorik	<u> </u>	(Modular Sensor System)	
/Nordic Sensor or Applied Sensors		NST 3210, NST 3220, NST 3220A, VOCmeter, VOC check	
		OEM Modules	
Aromascan /OSMETECH	http://www.aromascan.com/	Aroma Scanner	
WMA Airsense Analysentechnik Gmbh	http://www.airsense.com/uk/main.htm	PEN	
RST Rostock	http://www.rst-rostock.de/d/index_prod_sens.html http://www.rst-rostock.de/	SamSystem	
		SamDetect	
		SamDirect	
		SamSelect	
Bloodhound Sensors, Lltd.	www.leeds.ac.uk/ulis/bloodhound/frames/fr_home.htm	Bloodhound, BH114'	
Zellweger Analytics	http://www.detect-measure.com/z.htm http://www.detect-measure.com/default.htm	Search point optima	
		Searchlineexcel	
		705 sensor	
Neotronics Scientific Ltd.,		e-nose 4000	
		e-nose 5000	
HKR Sensorsysteme, GmBH		Olefactometer, Chemosensory system QMB-6, MS-Senosr, SensiTOF	
Mosaic Industries		Rhino	
Array Tec		Scan Master II	
Mastiff Electronic Systems		Scentinel	
Europhor Instruments		Alabaster-UV	
New Cosmos-Electric Co.		Portable odor level indicator, XP-329	
Sensidyne, Inc.,	http://www.sensidyne.com/	Portable odour level monitor	
National		Oral checker	
Marconni Applied Technologies	www.marconi-tecchnologies.co.uk	e-Nose 5000	
GSG-Analytical	http://gsg-analytical.com/enose.htm http://gsg-analytical.com/english/moses2.htm#	MOSES II	
Oligosense byba	http://chem-www.uia.ac.be/	Development	
Osmetech plc	http://www.osmetech.plc.uk/ http://www.osmetech.plc.uk/technology/techback.html	MultiSampler-SP, And Sensors, Core sensor module	
Smart Nose	http://www.smartnose.com/ http://www.smartnose.com/specificationsTechnique.htm	Smart Nose 300	
Element Ltd., or RKS Sensor Systems	http://www.element.is/	FreshSense	
Environics Industry Oy	http://www.uku.fi/laitokset/ympkem/Bio/imcell.html	MGD-1	
KHAMINA E-Nose	www.holzschuh-usa.com	KHAMINA Demonstrator	
		A A A A A A A A A A A A A A A A A A A	





Electronic nose sensor technologies

Sensor type	Principle of operation	Fabrication methods	Sensitivity
Metal oxide	Conductivity	Microfabricated	5-500 ppm
Conducting	Conductivity	Microfabrication	0.1 - 100 ppm
polymer		Electroplating	
		Screen printing	
Quartz crystal	Piezoelectricity	Screen printing	1 ng mass
microbalance		Wire bonding	change
		Microelectromecha	
		nical systems	
		(MEMS)	
Surface acoustic	Piezoelectricity	Microfabrication	1 pg mass
wave (SAW)		Screen printing	change
MOSFET	Capacitive charge	Microfabricatiojn	ppm
	coupling		
Gas	Molecular spectrum	MEMS, precision	Low ppb
Chromatography		machining	
Mass	Atomic mass	MEMS, precision	Low ppb
spectrometry	spectrum	machining	

JPL Reliability Issues





- Response reproducibility between sensors of nominally the same type. This is important for two reasons
- If a sensor is poisoned then it can be replaced without the need to fully recalibrate and retain the system.
- If the sensors are sufficiently reproducible in their response it becomes possible to train one sensor array and then use this same training set for any number of nominally identical arrays in other instruments at different locations.
- Sensitivity to changes in temperature, humidity, and flow rate are also important characteristics. Odour sensors should be insensitive to changes in temperature, humidity, and flow rate. Most sensors show some sensitivity problem.

JPL Reliability Issues





- These effects can be minimized by careful system design and sample handling which makes the instrument more complex, expensive, and limit sample throughput.
- In portable electronic noses it will be essential to overcome, or at least adequately compensate for, the effects of changes in temperature and humidity on the sensors baseline and on the magnitude of their responses.
- Sensor physical size need to be kept low. If the sensor is large then sample volume is large, which can lead to slow response time and poor sensitivity because of dilution.
- Microfabricated sensor array can meet these requirements.



Important instrument and test conditions when employing electronic nose technology

Parameter/Condition

Effect

Temperature

auses significant

Humidity

changes in all types Significant variation in

Reference gas

many sensors

Flow rate

Stable baseline signal, reduces interference

Odour concentration

Sensor output varies

Natural variations

cause errors in sensors

Odour profile

Repeatable

concentration profile

may improve

discrimination power



Package



- ◆ 32 Sensor head in an e-nose arrangement on 4 substrates each with 8 sensors
- ◆ Hybrid microelectronic co-fired ceramic alumina substrate
- ◆ Thick film screen printing
- ◆ A guard ring around each sensor
- ◆ 16 polymers used
- ◆ Two sensors for each polymer
- ◆ Four polymers on each substrate
- ◆ Thermistor is used to control temperature
- ◆ Gold contacts



Space Systems

- N
- Qualification of the devices for space systems
- Reliability
- Battery of test methods to establish above to validate the efficacy of E-Nose
- Variety of E-Nose configurations makes difficult in terms of qualification procedures
- Building in quality E-Nose



Space Systems



- Minimization of size and weight
- Improve performance
- Enhance the awareness of E-Nose to designer of space systems
- Advanced electronics packaging
- Radiation tolerant microcircuits

JPL Qualification





- ◆Inadequacy of military standards
- ◆Required amendment of standards
- ◆Risk assessment or factor
- ◆Assurance of new technologies

JPL Qualification Process

- **◆** Identification of technology failure modes
- ◆ Certainty of non-failure modes during the life of the mission
- ◆ Development of test methods for demonstrating failure modes (This is the current practice)
- **♦** Hermetic encapsulation
- **◆** Availability of Packaging support equipment
- ◆ Cost of packaging is a key acceptance criteria
- ◆ Space is one of the last place for the debut of a new technology

JPL

Environmental

- Conditions of storage
- Usage environment
- Qualification testing
- Incubation period of space electronics assemblies in various storage locations
- Thermal cycling
- Vibration due to launch and pyrotechnic release actions
- Hermetic packaging
- Outgassing products from polymeric materials



JPL Environmental (continued)

- Shock
- Humidity
- Radiation
- Corrosion
- Mismatches in TCE
- Extremes in ambient temperatures
- Thermomechanical shock



Issues to Address by Space Community



- New ways to designing systems
- Fully exploit the advantages of E-Nose and COTS E-Nose, ASIM technology
- Characterization of materials and systems at micron level
- Development of low-cost design, production, and test technologies
- Packaging and interconnection approaches
- Software interface
- Development of new product-assurance tools
- Versatile electronics package intended to drive/control microinstruments
- Software tools for data acquisition and analysis

Factors Influencing Package Reliability



- Residual stress
- Stress relief
- Hermeticity
- Thermal performance
- Chemical stability
- Protection during packaging
- Shock resistance
- Electrical isolation
- Cost issues



Package



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- ◆ Gold contacts

JPL Packaging Reliability Concerns

- * Typical mechanical fatigue
- ***** Contamination
- **E-Nose fatigue**
- Media compatibility
- Device passivation and Alternative chip mounting techniques







Future Prospects of E-Nose

- Likely to make many advances in the near future
- Presence of commercial instruments to stimulate interest
- Multitype noses
- Micro and nanotype e-noses
- ASIC Electronics
- Adaptive neural networks
- Smarter, smaller, and cheaper e-noses



Important instrument and test conditions when employing electronic nose technology

Parameter/Condition

Effect

Temperature

auses significant

Humidity

changes in all types Significant variation in

Reference gas

many sensors

Flow rate

Stable baseline signal, reduces interference

Odour concentration

Sensor output varies

Natural variations

Odour profile

cause errors in sensors

Repeatable

concentration profile

may improve

discrimination power

JPL <u>Conclusions</u>

- Assessing and evaluating the reliability of JPL and Commercial E-noses using the work-plan described for organic, inorganic and incipient fire detection.
- Thermal cycling and vibration tests of electronic packages such as driver/control electronics, e-noses under extreme temperatures will be performed for present and future NASA missions.
- ② Optical and non-destructive evaluation of electronic packages and e-noses before and after extreme cold temperature cycling.
- Prepare inspection criteria, develop protocols for flight experiment and shuttle crew, prepare quality assurance and reliability guidelines.
- Identified Four commercial E-Noses for evaluation of sensor head and respective IC packages
- Detected N₂H₄ at PPB level using KAMINA